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OPTICAL CHARACTERIZATION OF RARE EARTH DOPED WIDE BANDGAP SEMICONDUCTORS

FINAL PROGRESS REPORT

Uwe Hömmerich

August 6, 1999

U.S. ARMY RESEARCH OFFICE

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BODY OF REPORT

A. STATEMENT OF PROBLEM

Rare earth doped semiconductors are of significant current interest for possible applications as light emitting diodes (LED's), lasers, optical amplifiers, and full color displays. Major problems limiting the advancement of these devices are the weak rare earth luminescence efficiency and the low rare earth concentration in semiconducting hosts.

Among rare earth ions, trivalent Erbium has received most attention. It is well known that Er^{3+} ions exhibit atomic-like, temperature stable luminescence at 1.54 μ m due to the intra-4f-shell Er^{3+} transition ${}^4I_{13/2} \rightarrow {}^4I_{15/2}$. Since the 1.54 μ m emission from Er^{3+} overlaps the minimum absorption region of silica-based optical fibers, Er doped semiconductors are suitable light sources for optical communications. In contrast to existing 1.54 μ m sources, which are based on optically excited Er^{3+} ions in insulating materials or on the temperature sensitive bandedge emission from semiconductors, Er doped semiconductors offer the prospect of electrically pumped, compact, and temperature stable optoelectronic devices [1-3].

The photoluminescence (PL) properties of Er doped Silicon and several III-V materials have been extensively studied for more than a decade, however, only weak Er³⁺ PL was observed at room temperature [1-3]. It was first reported by Favennec et al. that the Er³⁺ PL intensity depends strongly on both, the bandgap energy of the semiconductor and the host temperature [4]. These researchers observed that the thermal quenching of Er³⁺ PL decreased for semiconductor hosts with larger bandgap. Therefore, current research activities focus on investigating Er³⁺ ions doped into wide-gap semiconductors [1-3].

The recent development of blue light emitting diodes and lasers based on GaN has stimulated an enormous amount of activity in the field of III-nitride semiconductor research [5]. The bandgap of III-nitrides ranges from 1.9 eV (InN) to 6.2 eV (AlN), which makes these materials also interesting candidates for Er³+ doping. Intense room temperature luminescence at 1.54 µm has been reported from Er doped GaN and AlN [6-13]. The first demonstration of electroluminescence from Er implanted GaN was reported by Torvik et al. in 1996 [13]. Subsequently, other researchers have reported infrared and visible electroluminescence from Er doped GaN. These results demonstrate that III-nitrides are suitable host for Er³+. However, the initial reports also indicated that much more spectroscopy and materials research is necessary to fully explore the incorporation and excitation mechanisms of Er³+ ions in III-nitrides. In this final report, spectroscopic and device studies of Er implanted GaN and in-situ Er doped GaN and AlN samples are presented.

B. SUMMARY OF MOST IMPORTANT RESEARCH RESULTS

The research results of this project have been discussed in great detail in 16 scientific publications as listed in section C. Reprints or preprints of these papers were provided to ARO during the course of the work. In the following the most important research results are summarized:

1. Er implanted GaN

During the course of this project, extensive photoluminescence (PL) and photoluminescence excitation (PLE) study were carried out on Er implanted GaN. The GaN film was grown on a sapphire substrate using reactive ion-beam MBE [6]. Er was implanted at an energy of 300 keV and a fluence of $2x10^{14}$ cm⁻². Oxygen was co-implanted at an energy of 40 keV and a fluence of 10^{15} cm⁻². After implantation the sample was annealed at ~650° C for approximately 60 minutes. After optical excitation at 325 nm and 488 nm, strong 1.54 μ m Er³⁺ PL was observed, which remained temperature stable from 15 to 575 K (see figure 1). At 575 K, the integrated PL intensity decreased by ~10% for above gap excitation (λ_{ex} =325nm) and ~50% for below gap excitation (λ_{ex} =488nm) relative to its value at 15 K. To our knowledge, the investigated GaN: Er sample exhibits the weakest PL temperature quenching observed from any Er³⁺ doped III-V semiconductor to date, even less than Er³⁺ doped SiC [2]. The excellent temperature stability makes GaN: Er very attractive for high temperature opto-electronic device applications.

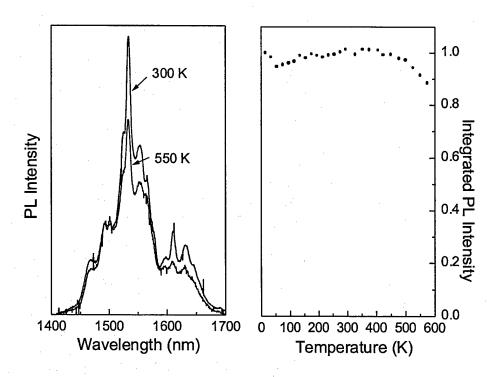
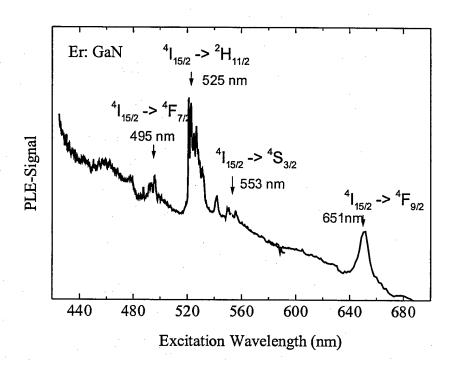


Figure 1: Photoluminescene spectra of Er implanted GaN at 300 and 550K. The integrated luminescence intensity remained nearly constant up to 550K.

Photoluminescence excitation (PLE) measurements were conducted to gain insight in the Er³⁺ excitation mechanisms in the GaN host (see figure 2). The PLE results show that Er³⁺ can be excited continuously over a broad wavelength region spanning from 425 to 680 nm. This observation provided the first experimental evidence that defects in GaN play a significant role in the Er³⁺ excitation process. In addition to the broad PLE band, sharp PLE features were observed at approximately 495 nm, 525 nm, 553 nm, 651 nm, and 980 nm and they were assigned to intra-4f transitions of Er³⁺ ions. Overall, the PLE spectrum shown in figure 2 indicates that optically active Er³⁺ ions can be excited either through carrier-mediated processes involving defects in the host or through resonant pumping into Er³⁺ 4f energy levels. With respect to these two excitation schemes, distinct Er³⁺ PL properties were observed for resonant and off-resonant Er³⁺ excitation indicating the presence of different subsets of Er³⁺ ions in GaN.



Photoluminescence excitation spectrum of Er implanted GaN. The broad PLE band spanning from 425 to 680 nm was attributed to indirect, carrier-mediated Er excitation. The sharp features at 495, 525, 553, 651, and 980 nm were assigned to intra 4-f Er³⁺ transitions.

3. Er doped AlN prepared by metalorganic molecular beam epitaxy (MOMBE)

Doping Er through ion implantation has severe disadvantages for device applications because it limits the depth and distribution of Er³⁺ ions and can cause significant damage to the host material. Therefore, the goal of current Er doped Nitride research is to incorporate Er³⁺ ions during epitaxial growth. In-situ Er doping of AlN using metalorganic molecular beam expitaxy (MOMBE) was first

reported by MacKenzie et al. in 1996 [8]. In the following, material preparation and spectroscopic results of these AlN: Er samples are summarized.

Materials:

The sample used in this study was prepared at the University of Florida [8]. The AlN film was grown by MOMBE in an INTEVAC Gas Source Gen II on 2" (0001) Al₂O₃. The film was preceded by a low temperature AlN buffer (Tg 425°C). Dimethylethylamine alane (DMEAA) was used as the Al source. A reactive nitrogen flux was provided by a Wavemate MPDR 610 electron cyclotron resonance (ECR) plasma source with an Al₂O₃ cup mounted directly in the growth chamber source flange. A shuttered effusion oven charged with 4N Er was used for erbium doping. Er incorporation was profiled by secondary ion mass spectrometry (SIMS). The Er concentration was determined to be 2-5×10¹⁹ Er/cm³.

Spectroscopic Results:

Strong 1.54 μ m Er³⁺ was observed from AlN: Er (MOMBE) samples with intensities nearly two orders of magnitude greater than those observed in Er implanted AlN samples [6]. Thermal quenching experiments were carried out (see figure 3) and indicated that the Er³⁺ PL intensity decreased by only a factor of 2 between 15 and 300 K.

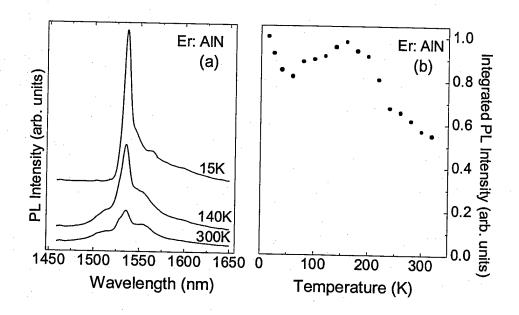


Figure 3: (a) Er PL spectra for AlN: Er at three different temperatures (b) Temperature dependence of the integrated Er PL for AlN: Er.

The PLE spectrum of Er^{3+} in Er: AlN at 15 K is depicted in Fig. 4. In this experiment, the Er^{3+} luminescence was monitored at 1.54 μm while varying the excitation wavelength. Similar to Er implanted GaN, the PLE spectrum featured a broad band starting at ~ 600 nm and extending to 420 nm, the short wavelength limit of the OPO system. Superimposed on the broad band were a number

of sharp features located at 456, 494, 525 and 653 nm, suggesting a direct optical excitation of 4f energy levels of Er^{3+} . Tentatively, we attribute these peaks to the following intra-4f Er^{3+} transitions: ${}^4I_{15/2} \rightarrow {}^4F_{3/2,5/2}$, ${}^4I_{15/2} \rightarrow {}^4F_{7/2}$, ${}^4I_{15/2} \rightarrow {}^2H_{11/2}$, and ${}^4I_{15/2} \rightarrow {}^4F_{9/2}$, respectively. The broadband excitation is most likely due to carrier-mediated processes involving Er-related levels, analogous to the below-bandgap excitation of Er^{3+} in Er implanted GaN.

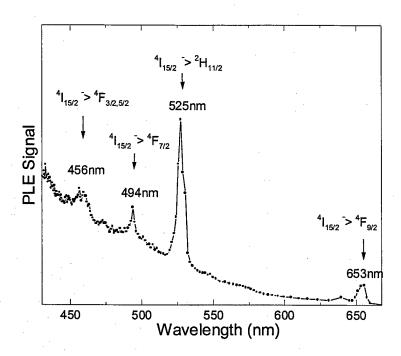


Figure 4: Photoluminescence excitation spectrum of AlN: Er.

To draw more information on the two types of Er3+ excitation processes, time-resolved PL measurements excited at various wavelengths were made. In Fig. 5 (a, b, and c) the low temperature (15 K) Er³⁺ PL decays are shown excited at the intra-4f Er³⁺ absorption peaks (494, 525, and 653 nm) and at the vicinity of the peaks (497, 537, and 640 nm). The Er³⁺ PL decay transients were found to be non-exponential at all excitation wavelengths. Following a fast initial decay (lifetime ~50 µs), the Er³⁺ PL decayed with a long lifetime of ~0.83 ms. It was noted that Er³⁺ PL decays excited at the intra-4f absorption peaks were longer-lived, containing less fast-decay component than those excited through a carrier-mediated process at the wavelengths not matching the intra-4f transitions. The discrepancies between Er3+ PL decays excited at 640 and 653 nm, 525 and 537 nm were especially pronounced. On the other hand, the Er³⁺ PL decay curve excited at 494 nm differed only slightly from that excited at 497 nm, very likely due to the relatively weak transition ${}^{4}I_{15/2} \rightarrow {}^{4}F_{7/2}$ compared to the carrier mediated contribution. The de-excitation of Er3+ PL depends on nonradiative quenching processes associated with the specific local Er3+ environment. Therefore, the different appearance between the Er3+ PL decays excited through carrier-mediated processes and direct optical intra-4f excitation suggests that two different subsets of Er3+ sites are excited in respect to the two types of excitation mechanisms. At least two Er³⁺ centers, one long-lived and other shortlived Er³⁺ centers, are formed in Er: AlN. The long-lived Er³⁺ sites are excited primarily via direct intra-4f excitation of Er³⁺ and the short-lived through indirect carrier-mediated processes.

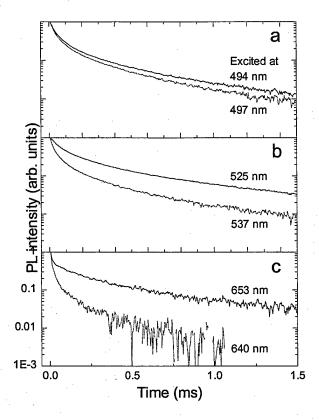


Figure 5: Er PL decay curves for AlN: Er following pulsed excitation at different wavelengths.

The data show that at least two classes of Er ions with distinct PL behavior are present in AlN.

2. Er doped GaN prepared by metalorganic molecular beam epitaxy (MOMBE)

The successful in-situ Er doping of ALN has prompted interest in doping GaN by MOMBE technique. The first in-situ doped GaN: Er samples were prepared in 1997 by MacKenzie et al. [7,8]

Materials:

The investigated Er doped GaN films were provided by the University of Florida [7]. All samples were grown by metalorganic molecular beam epitaxy (MOMBE) in an INTEVAC Gas Source Gen II on In-mounted (100) Silicon or (0001) Sapphire substrates. The GaN films were preceded by a low temperature AlN buffer (T_g=425 °C). A 0.2 µm undoped GaN spacer was deposited prior to the growth of GaN: Er. Triethylgallium (TEGa) and Dimethylethylamine Alane (DMEAA) provided the group III fluxes. A shuttered effusion oven with 4N Er was used for solid source doping. Reactive nitrogen species were provided by a SVT radio frequency plasma source. Due to the incorporation of carbon and oxygen from residual ether in TEGa, the C and O

background observed were $\sim 10^{21} \text{cm}^{-3}$ and $\sim 10^{20}$ cm⁻³, respectively, as determined by SIMS measurements.

Spectroscopic Results:

During the course of this project extensive spectroscopic studies were carried out on in-situ Er doped GaN. The measurements included spectral and intensity measurements as a function of oxygen and carbon concentration, spectral and intensity studies under different excitation conditions, PL quenching and lifetime studies, pump power dependence luminescence studies, and photoluminescence excitation studies.

i) PL studies of GaN: Er for different Carbon and Oxygen background concentrations

Figure 6 shows an overview of the infrared PL spectra of Er doped GaN on Al₂O₃ prepared by MOMBE. No significant spectral differences were observed for Er doped GaN on Sapphire or Si substrates. For comparison, the Er3+ PL spectra of AlN: Er (MOMBE) and Er implanted GaN are also shown in figure 6. All samples were excited with the 363-366.8nm UV output of an Argon-Ion laser. UV excitation was chosen to simulate carrier-mediated Er3+ excitation as in GaN: Er electroluminescence devices. All samples showed characteristic Er3+ PL centered at 1.54 µm, which can be assigned to the intra-4f $\mathrm{Er^{3+}}$ transition ${}^{4}\mathrm{I}_{13/2}$ ---> ${}^{4}\mathrm{I}_{15/2}$. The $\mathrm{Er^{3+}}$ PL spectra from in-situ Er doped GaN and AlN are very similar. They exhibit nearly featureless spectra (25-35nm @FWHM) suggesting a homogenous distribution of Er sites as typically observed from Er doped glasses. Er peaks located at 1.517 μm, 1.540 μm, 1.548 μm, and 1.555 μm are either due to Stark splittings and/or multiple Er sites. The overall spectral width of the Er3+ PL from Er implanted GaN was significantly larger (100nm@FWHM) than that observed from Er doped GaN and AlN. Moreover, Er implanted GaN exhibits a more sharply structured Er³⁺ PL than the in-situ Er doped III-Nitride samples. Based on previously published data, the more complex and broad Er3+ PL seems to be characteristic for Er implanted GaN samples and is due to a combination of different Er sites and Stark splittings of Er ions in low symmetry sites.

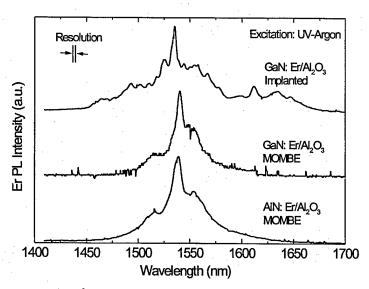


Figure 6: Room-temperature Er³⁺ PL spectra of Er implanted GaN and in-situ Er doped GaN and AlN.

Figure 7 depicts a comparison of the Er³+ PL intensities of GaN: Er (MOMBE) for below and above-gap excitation at room-temperature. Under below-gap excitation, GaN: Er (TEGa) with high oxygen and carbon background ([O]~10²0cm⁻³, [C]~10²1cm⁻³) showed an Er³+ PL peak intensity two orders of magnitude larger than GaN: Er (Ga) with low O and C backgrounds (<10¹9cm⁻³). It is interesting to note that also GaN: Er/Si showed strong 1.54 μm PL at room temperature, which makes this material attractive for integration with Si based optoelectronics. Drastic improvements of the absolute Er³+ PL intensity have been observed for Er and oxygen codoped Si and GaAs [1,2].

The enhanced 1.54 μm PL was attributed to an increased concentration of optically active Er³⁺ ions and a more efficient Er³⁺ PL excitation. Our results for below-gap Er³⁺ excitation in GaN suggest that the incorporation of O and C introduces beneficial mid-gap states that provide efficient energy transfer pathways for Er³⁺ in GaN. Support for this idea was obtained from photoluminescence excitation (PLE) studies shown in figure 8. In PLE the effectiveness of stimulating 1.54 μm Er³⁺ PL is measured as a function of excitation wavelength. The PLE spectra of GaN: Er/Si (TEGa) reveal that the incorporation of high O and C backgrounds leads to a broad PLE band extending over the entire visible region (~400-800 nm). The GaN: Er (Ga) sample with low C and O content did not show this broad PLE band, but direct intra-4f Er transitions (see e.g. ~525nm). Therefore, the visible output from an Argon-Ion laser leads to a significantly more efficient excitation of Er³⁺ in GaN: Er/Si (TEGa) than in GaN: Er/Si (Ga).

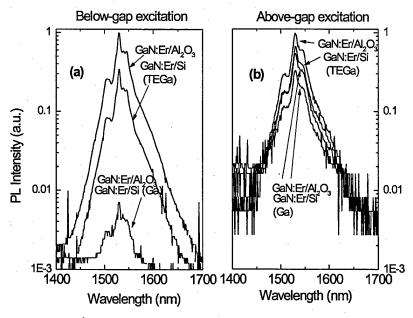


Figure 7: Comparison of Er³⁺ PL intensity from Er doped GaN (MOMBE) for (a) below-gap and (b) above-gap excitation (300K).

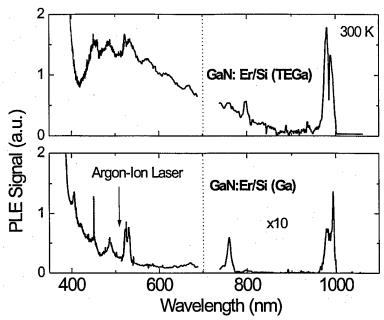


Figure 8: PLE of GaN: Er/Si samples with varying oxygen and carbon content. The incorporation of high oxygen and carbon backgrounds (upper graph) leads to a broad Er³⁺ excitation band covering the entire visible region.

Under above-gap excitation (figure 7b) all GaN: Er (MOMBE) samples showed a greatly reduced Er³⁺ PL intensity relative to below-gap excitation, independent of carbon and oxygen concentration. For example for GaN: Er/Sapphire the peak Er PL intensity at 1.54µm decreased by roughly a factor of ~55 when changing the pump wavelength from 442 nm to 325nm. A similar PL reduction was reported for Er implanted GaN. It is not yet clear why the Er³⁺ PL intensity was so greatly reduced under above-gap excitation. Preliminary visible luminescence studies indicate that the bandedge provides another radiative recombination channel, which reduces the overall Er³⁺ excitation efficiency. More work is still in progress to elucidate the correlation between infrared Er³⁺ PL intensity and near bandedge luminescence from Er doped GaN.

ii) Pump Intensity Dependent Photoluminescence Study

The Er³+ PL intensity of GaN:Er/Si with high oxygen and carbon background was studied as a function of pump intensity in order to gain more insight in the Er³+ PL properties. Figure 9a) shows the Er³+ PL intensity monitored at 1.54 μm as a function of pump intensity at room temperature under above and below-gap excitation. It can be noticed that under below-gap excitation the Er³+ PL intensity shows a nonlinear behavior suggesting the onset of Er³+ PL saturation. Contrary, under above-gap excitation a nearly linear increase of the Er³+ PL intensity was observed up to pump intensities as high as ~100W/cm². The linear increase indicates that only a fraction of Er ions has been excited, even at the high pump intensities.

The observed pump intensity behavior can be modeled using a simple three level Er^{3+} energy scheme. The excitation of Er^{3+} into a pump level occurs with an absorption cross-section (σ_{abs}) and

is followed by a fast relaxation into the metastable level. Only a certain fraction (η_p) of the pump level population quickly ends in the metastable level. The product of absorption cross section (σ_{abs}) and pump efficiency (η_p) is used in the following as the overall Er^{3+} excitation efficiency (σ_{ex}) . After balancing the rates of excitation and deexcitation processes under steady state pumping, it can be derived that the pump power dependence of the Er^{3+} PL intensity is given as:

$$I = \frac{N}{\left(1 + \frac{hc}{\tau \cdot \sigma_{ex} \cdot P \cdot \lambda_{p}}\right)} \cdot w_{r} \cdot C \tag{1}$$

where N is the concentration of optically active Er^{3+} ions, w_r is the radiative decay rate, C is the collection efficiency of the luminescence setup, τ is the luminescence lifetime, σ_{ex} is the excitation efficiency, λ_p is the excitation wavelength, and P is the pump intensity. In order to model the data shown in figure 9a), we assumed that the collection efficiency of the PL setup, the number of optically active Er^{3+} ions, and the radiative decay rate are independent of excitation wavelength. The luminescence transients for above (λ_{ex} =355 nm) and below-gap (λ_{ex} =532 nm) excitation are depicted in figure 9b). For comparison, the system response is also shown in Figure 9b).

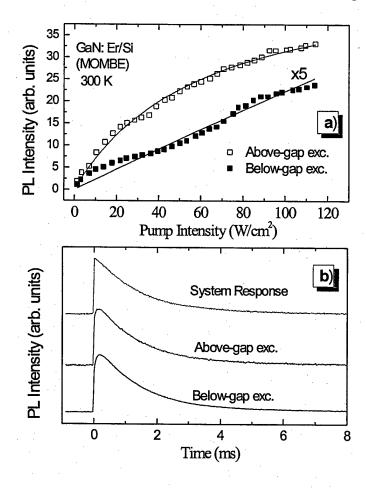


Figure 9: a) Er³⁺ PL intensity of GaN:Er/Si as a function of pump intensity. A similar pump intensity behavior was observed for GaN: Er/Sapphire. The solid line is the best fit to equation (1). b) Decay transients of GaN:Er/Si under above and below-gap excitation.

Within the experimental error of the measurement we observed that the lifetime does not dependent on the excitation wavelength. The only remaining parameter used to fit the pump intensity data shown in figure 9a) to equation 1 was the excitation efficiency. The obtained best-fits revealed that the excitation efficiency under above-gap pumping is reduced by a factor of ~30 compared to below-gap excitation. As discussed before, preliminary visible luminescence studies have shown, that the bandedge provides another radiative recombination channel, which reduces the overall Er³⁺ excitation efficiency under above-gap excitation. To what extend this reduction in excitation efficiency is due to a wavelength dependent absorption cross-section and/or pump efficiency is not yet known.

Development of a Hybrid 1.54 µm GaN:Er/InGaN LED

The development of blue/green InGaN LED's has made possible novel optoelectronic applications involving wavelength down conversion. These devices can be classified as "hybrid LED's" because they are based on the optical excitation of light emitting centers using the electroluminescence from InGaN. A white light source was demonstrated by Nakamura using an InGaN LED to optically pump organic phosphor materials [5]. Another example of achieving white light emission was recently reported by Hide et al. [14]. These authors used an InGaN LED to optically excite conjugated polymers. Depending on the polymer film, the emission of this hybrid InGaN/conjugated polymer LED was tunable across the chromaticity diagram. As discussed before, our current GaN: Er films showed a significantly larger Er³⁺ excitation efficiency for below-gap excitation than for above-gap excitation. This observation has stimulated our interest in developing a hybrid LED based on Er doped GaN.

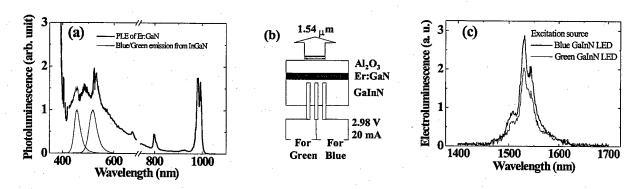


Figure 10. (a) The blue/green emission from a GaInN LED overlaps the broad-band absorption in GaN:Er (b) design of the hybrid InGaN/GaN:Er LED (c) 1.54-μm infrared luminescence from the device shown in (b).

Using PLE spectroscopy, we investigated the below-gap absorption bands of Er³⁺ in more detail and compared the result to the light emission from a commercial blue/green InGaN LED. As mentioned before (see also Figure 10a) Er³⁺ ions in GaN with high oxygen and carbon background concentrations can be excited continuously at any wavelength between 400-600 nm. The broad Er³⁺ absorption band nicely overlaps the output of an InGaN LED with emission bands peaking at 450 nm and 520 nm as shown in the lower trace of Figure 10a). These measurements indicate the possibility of a hybrid LED in which an Er doped GaN film is optically excited by a commercial

blue/green InGaN LED to produce emission at 1.54 µm. Figure 10b) shows the design of a hybrid GaN:Er/InGaN LED and its operation. In this experiment a blue/green Nichia InGaN LED was employed to excite the Er³+ ions in the GaN layer grown on a sapphire substrate. The epoxy bubble of the InGaN LED was removed and polished. The GaN: Er film was placed directly on top of this LED as shown in figure 10b). The DC forward voltage and current of the InGaN LED were 2.98 V and 20 mA, respectively. The luminescence at 1.54 µm under blue or green pumping was collected through the sapphire substrate and is displayed in figure 10c). Even though the resulting emission at 1.54µm was weak, it demonstrates that the electroluminescence from InGaN can be used to excited Er³+ ions in GaN.

Summary

During the funding period of this grant we have carried out extensive spectroscopic studies on Er implanted GaN and in-situ Er doped AlN and GaN prepared by MOMBE. In each case strong 1.54 µm luminescence was observed at room temperature. However, the incorporation and excitation schemes of Er in III-Nitrides is rather complex and strongly dependent on the material synthesis. For example, it was found that high oxygen and carbon concentrations (~10²⁰ cm⁻³) in GaN: Er samples greatly enhance the 1.54 um Er³⁺ PL intensity under below-gap excitation. Moreover, PLE studies revealed that oxygen/carbon introduces a broad below-gap PLE band, which provides an efficient pathway for Er³⁺ excitation. Under above-gap excitation, the absolute Er³⁺ PL intensity from GaN: Er (MOMBE) samples was greatly reduced and only a weak PL dependence on the oxygen and carbon concentration was observed. Pump intensity dependent Er3+ PL studies of GaN:Er/Si revealed that the Er³⁺ excitation efficiency for above-gap pumping is reduced by a factor of ~30 compared to below-gap pumping. The low above-gap excitation efficiency will be a limiting factor in current electroluminescence devices grown by MOMBE and employing forward-biased carrier injection. Based on the efficient below-gap Er excitation a novel hybrid InGaN/GaN: Er LED operating at 1.54 µm has been demonstrated. Questions concerning the overall efficiency of this novel hybrid LED need to be further addressed in the future. More materials engineering will be necessary to optimize the 1.54µm Er³⁺ PL from GaN: Er for practical device applications.

C. LIST OF PUBLICATIONS AND TECHNICAL REPORTS

PUBLICATIONS:

- U. Hömmerich, J. T. Seo, Myo Thaik, J. D. MacKenzie, C. R. Abernathy, S. J. Pearton, R. G. Wilson, J. M. Zavada, "Optical characterization of erbium doped III-nitrides prepared by metalorganic molecular beam epitaxy", Internet J. Nitride Semicond. Res. 4S1, G11.6 (1999).
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Graduate Student:

Myo Thaik:

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Athea Bluiett:

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Undergraduate Student:

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Kirk Peterson:

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INVENTIONS

none

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